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The fate of sulphur during oxy-fuel combustion of lignite

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Abstract

This work focuses on the sulphur chemistry in oxy-fuel combustion of lignite. Comparison is made with air-fired conditions, and the main combustion products containing sulphur (ash, SO_2, SO_3 and H_2S) are examined by modelling and experimental data. The conversion of fuel-S to SO_2 is lower in oxy-fuel than in air-fuel conditions and the SO_2 concentration per unit energy supplied is therefore reduced. The modelling shows that the SO_3 concentration is about four times greater than the concentration in air-firing. High SO_3 and H_2O contents in the oxy-fuel flue gas during wet recycling increase the acid dew-point by 20 to 30K.

Keywords: oxy-fuel; sulphur; SO_2; SO_3; ash

1. Introduction

In oxy-fuel (O_2/CO_2) combustion, oxygen is used instead of air to oxidise the fuel. The basic principle of oxy-fuel combustion is shown in Figure 1. Typically, flue gas is recycled to moderate the flame temperature, and the reactions take place in an atmosphere with elevated concentration of combustion products and low content of N_2. This has important implications for the combustion chemistry; here, some aspects of the sulphur-related chemistry will be examined.

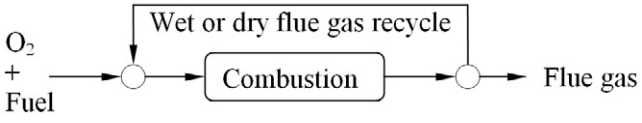

Figure 1. The basic principle of oxy-fuel combustion.

The aim of the work is to investigate the fate of sulphur in oxy-fuel combustion of lignite and especially to elucidate the differences in sulphur behaviour between air-fired and oxy-fuel conditions. Previous work on the

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sulphur chemistry in oxy-fuel combustion is briefly reviewed. Sulphur sinks and their impact on the process are evaluated and some aspects of the gas-phase chemistry are examined by modelling.

Figure 2 presents the main reaction routes of sulphur during combustion of coal. Sulphur is introduced by the coal, whose content of sulphur can vary from around 0.2 wt% up to 11 wt% [1,2]. Normally, the content is in the range of 0.5 to 4 wt% [3]. Sulphur in coal is found in the form of sulphides, organic sulphur compounds, sulphates and traces of elemental sulphur [4]. The sulphur content and the way the sulphur is bound vary with coal type and depend on age and location of the coal source [1]. The main part of the fuel-bound sulphur is released to the gas phase during combustion. Sulphides in the form of pyrite are usually a main fraction [2,4] released according to the reactions [5]:

$$2\ FeS_2\ (s) \rightarrow 2\ FeS\ (s) + S_2\ (g) \quad (1)$$

$$2\ FeS\ (l) \rightarrow 2\ Fe + S_2 \quad (2)$$

$$S_2 + 2\ O_2 \rightarrow 2\ SO_2 \quad (3)$$

Another important fraction of coal-S is organically bound sulphur [1,2,4,6]. The quantities of sulphates in coal are normally low [1,2]. Sulphur dioxide (SO_2) is the thermodynamically favoured sulphur oxide at high temperature (>1000°C) and oxygen-rich conditions. Under sub-stoichiometric conditions in the flame, hydrogen sulphide (H_2S) is formed in the gas or released from organic sulphur compounds. The equilibrium favours SO_2 at higher oxygen concentration and temperatures. Thus, if oxygen is in excess with quantities typical for pulverized coal combustors, the outlet concentration of H_2S is negligible. At lower temperatures, the equilibrium shifts towards sulphur trioxide (SO_3), but the reaction rate decreases with temperature and the concentration of SO_3 is several orders of magnitude lower than that of SO_2 in the emitted gas. In air-fired pulverised-coal combustion, typically 0.1 to 1% of the SO_2 is oxidized to SO_3 [7]. The following reactions are important for SO_3 formation:

$$SO_2 + O \rightarrow SO_3 \quad (4)$$

$$SO_2 + OH \rightarrow HOSO_2 \quad (5)$$

$$HOSO_2 + O_2 \rightarrow SO_3 + HO_2 \quad (6)$$

The formation of SO_3 depends on concentration of SO_2, residence time, temperature profile, concentration of O_2, fly ash composition, concentration of NO_2 and presence of catalysts (e.g. Fe_2O_3 [8]). At temperatures below 500°C, SO_3 reacts with H_2O in the flue gas to form gaseous H_2SO_4, which can cause corrosion if it condenses on metal surfaces or particles. SO_3 promotes particle formation, which is utilized to improve the performance of electrostatic precipitators, but it also increases plugging of air-preheater passages and emission of aerosols [9].

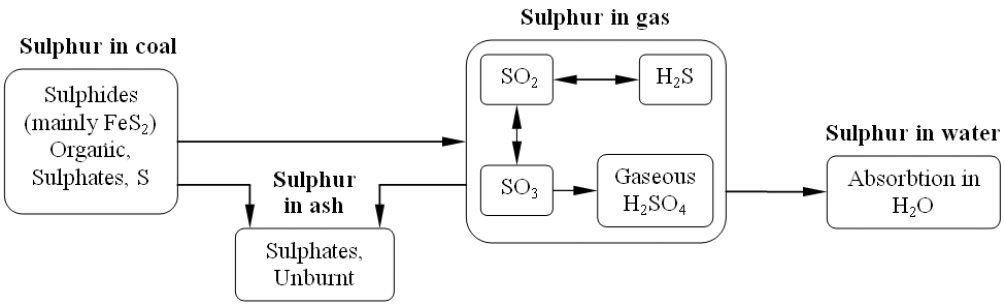

Figure 2. Main reaction routes of sulphur during combustion of coal.

Besides forming gaseous sulphur products, sulphur is captured by alkali and alkaline earth metals (Na, K, Mg, Ca) in the ash or is bound in the minerals without being released. Ca has a dominant role in sulphur retention; the Ca/S molar ratio in the coal is one of the main characteristics that governs the retention of sulphur in the ash [3,10].

If Ca is present in the coal or added as calcite ($CaCO_3$) or dolomite ($CaMg(CO_3)_2$), and the temperature is suitable, lime (CaO) is rapidly formed. CaO can then react with SO_2 to form $CaSO_4$ [6,10-12]:

$$CaCO_3 \rightarrow CaO + CO_2 \quad (7)$$

$$2\ CaO + 2\ SO_2 + O_2 \rightarrow 2\ CaSO_4 \quad (8)$$

Calcination (Reaction 7) is inhibited in oxy-fuel combustion at atmospheric pressure and temperatures below 890°C (~100K lower in air combustion), due to the high CO_2 partial pressure and direct sulphation of $CaCO_3$ is favoured (similar to the results obtained from pressurized air-combustion [13]):

$$2\ CaCO_3 + 2\ SO_2 + O_2 \rightarrow 2\ CaSO_4 + 2\ CO_2 \quad (9)$$

However at higher temperatures, common in pulverized coal combustion, calcination takes place [14]. Chen et al. [15] measured a larger pore diameter of CaO calcined in an oxy-fuel atmosphere than that calcined in an air atmosphere. The larger pore diameter yields a better sulphation of CaO due to reduced pore filling and plugging. $CaSO_4$ may dissociate at temperatures higher than 850°C, depending on the carbonate fraction [3] and the composition of the surrounding atmosphere. However the higher concentration of SO_2 in oxy-fuel combustion stabilizes the formed $CaSO_4$ [11,13,16]. Hence, the desulphurization efficiency of oxy-fuel combustion is significantly increased compared to air-firing [16,17]. Sulphur retention also depends on fuel-specific characteristics, such as coal particle size and the way sulphur, alkali and alkaline earth metals are bound [3,17]. SO_3 can also be captured by the ash, and at low temperatures, adsorption of SO_3/H_2SO_4 by particles is efficient. During combustion of sub-bituminous coals, commonly having a low sulphur content, the alkalinity of the fly ash is often high enough to adsorb nearly all H_2SO_4 (g) [9]. After combustion, sulphur compounds in the flue gas can be absorbed in condensing water, but this is not further investigated in this work.

2. Method

The main sulphurous combustion products evaluated in the present work are sulphur in the ash and in the gases: the latter being SO_2, SO_3 and H_2S. The proximate and ultimate analyses of the fuel (Lausitz lignite) are given in Table 1. The molar ratios of Ca/S, Mg/S, 2K/S and 2Na/S are 1, 0.5, <0.005 and <0.005.

Table 1. Analysis of the Lausitz lignite, LHV = lower heating value.

[MJ/kg]	Proximate [wt. %, as received]			[wt. %, d.a.f.]	Ultimate [wt. %, d.a.f.]				
LHV (as received)	Moisture	Ash	Combustibles	Volatile matter	C	H	N	S	O
21.1	10.4	5.5	84.1	56.9	67.0	5.5	0.8	0.9	25.9

The impact of sulphur retention and its efficiency depending on the combustion conditions are discussed in Section 3.1. Laboratory analyses have been carried out to determine the release of coal-S to SO_2 at different temperatures. A small lignite sample was burned in an oven (LECO SC 144) under a pure oxygen atmosphere at constant temperature, and the SO_2 content of the flue gas was measured (IR cell). Furthermore, data are compiled from some published experimental work to illustrate the conversion of coal-S to SO_2 under oxy-fuel and air-fired conditions. The sulphur retention during combustion of Lausitz lignite is calculated at different assumed sulphur contents in the ash. The sulphur retention is the fraction of coal-S retained in the ashes

$$Retention = (Y_{S,\,ash} \bullet Y_{ash,\,coal}) / Y_{S,\,coal} \quad (10)$$

where $Y_{S,\,ash}$ is mass fraction of sulphur in the ash, $Y_{ash,\,coal}$ mass fraction of ash related to the coal, $Y_{S,\,coal}$ mass fraction of sulphur in the coal. The respective SO_2 content in the flue gas if all other fuel-S is converted to SO_2 is calculated for complete combustion with CO_2, H_2O, SO_2, N_2 and 4% O_2 in the flue gas.

The gas-phase reactions and the formation of SO_3 and H_2S, investigated by means of a combustion model, are discussed in Section 3.2 and 3.4. The model assumes plug flow and uses a predefined temperature profile. Initially, the gas is assumed to be at its equilibrium composition after complete combustion of the lignite (Table 1) at the peak temperature. A similar approach was used by Srivastava et al. [9]. Three cases are investigated: air-firing, oxy-fuel

combustion with dry recycle, and oxy-fuel combustion with wet recycle, by varying the oxidizer as presented in Table 2. The gas phase reactions are modelled with a detailed chemical kinetic scheme together with the associated thermodynamic library based on the work of Alzueta et al. [18] with the updates presented by Rasmussen et al. [19]. The calculations are performed with the SENKIN [20] code and the EQUIL [21] code of the CHEMKIN-II [22] software. In the gas-phase model heterogeneous interactions are neglected, the most important one being retention of sulphur in ash.

Table 2. Composition of the oxidizers used in the calculations, RFG = recycled flue gas.

Component	Air	O_2/RFG_{dry}	O_2/RFG_{wet}
O_2	21%	29%	29%
N_2	79%	0.5%	0.5%
CO_2	0	69.5%	48.0%
H_2O	0	0.9%	22.4%
SO_2	0	760 ppm	760 ppm
NO	0	240 ppm	240 ppm

An acid dew-point calculation of a flue gas with different water contents is presented in Section 3.3. The acid dew-point temperature is calculated after Verhoff and Banchero [23].

3. Results and Discussion

The results are presented in four parts: sulphur retention and concentration of SO_2, formation of SO_3, acid dew-point and H_2S. The relative importance of the results and their impact on the oxy-fuel process are discussed.

3.1. Sulphur retention and sulphur dioxide

Figure 3 shows the release of coal-S to SO_2 from the lignite at different temperatures under laboratory conditions. The results match those of an investigation by Kasbohm [3], which included over 40 lignite samples. According to a standard method (SS 187177) it is assumed that all sulphur present in the coal is released as SO_2 at 1400˚C. The sulphur release is closely related to temperature, and at 1200˚C, for example, only 76% of the total sulphur is released. Below 600˚C the dominant sources of sulphur release are organically bound sulphur and pyrite. Thus, the combustion temperature is critical to the sulphur release.

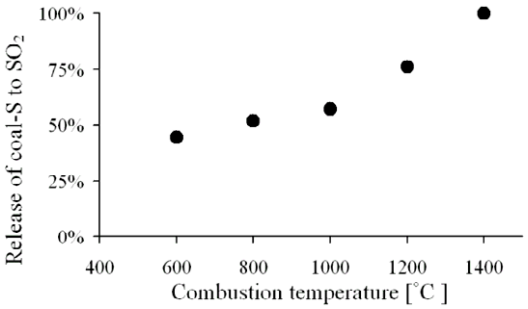

Figure 3. SO_2 release from Lausitz lignite at different temperatures under laboratory conditions.

Figure 4 shows the conversion of coal-S to SO_2 under oxy-fuel and air-fired conditions measured in some published work [24-29]. It is obvious that the conversion of coal-S to SO_2 is lower in the oxy-fuel case compared to air-fired conditions. However, the SO_2 concentration in the flue gas is much higher in oxy-fuel combustion due to the recycling of SO_2 and the higher O_2 content in the flue gas (lower flue gas volume). For example, Croiset and Thambimuthu [25] measured a outlet concentration of around 600 ppm SO_2 in air-firing and around 1600 ppm in average for oxy-fuel combustion (fuel: bituminous coal with 0.96% S).

Part of the SO_x formed is captured and bound to inorganic compounds in the ashes downstream of the combustor. Another part of the fuel-S is still bound in the minerals without being released. Figure 5 shows the sulphur retention for different sulphur contents in ashes for combustion of Lausitz lignite together with the calculated remaining SO_2 in the flue gas under oxy-fuel conditions. For example, with a sulphur content of 9% in the ash, the sulphur retention would be 60% and nearly 2000 ppm SO_2 would be measured in the flue gas. In principle, Lausitz lignite contains enough Ca to capture all sulphur as $CaSO_4$. However, all Ca is not available to capture sulphur, since Ca is also bound in inactive constellations like, for example, silicate minerals.

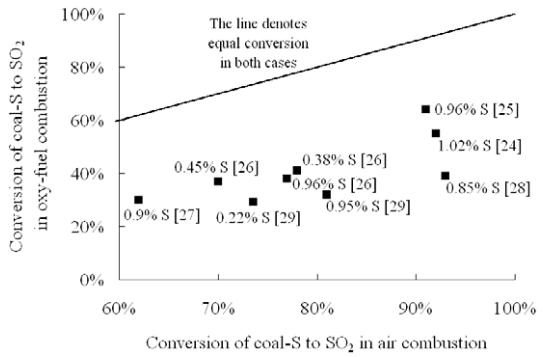

Figure 4. Conversion of coal-S to SO_2 in oxy-fuel and air combustion from some published experimental work at various sulphur contents.

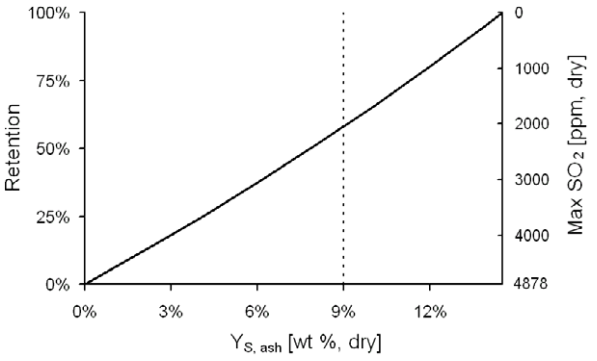

Figure 5. Sulphur retention in the ashes according Eq. 10 and remaining SO_2 concentration in the flue gas during oxy-fuel combustion of Lausitz lignite. The dashed line is an example.

If the amount of SO_3, H_2S and the absorption of sulphur compounds in condensing water is negligible the retention of sulphur in ash can be calculated by the conversion of coal-S to SO_2:

$$Retention = 100\% - Conversion \quad (11)$$

3.2. Sulphur trioxide

The formation of SO_3 is investigated for the three cases of boiler operation shown in Table 2: air-firing and oxy-fuel combustion with wet and dry recycle. Figure 6 shows a comparison between the SO_3 formation in oxy-fuel combustion with dry recycle and air combustion. The additional SO_3 formed during wet recycle is negligible (~3 ppm compared to dry recycle) and is therefore not shown. The outlet concentration of SO_3 in oxy-fuel combustion is about four times the concentration in air-firing when the gas follows a predefined temperature profile typical for a coal-fired power plant [30]. SO_3 is primarily formed during the cooling of the flue gas in the temperature range around 1300°C to 900°C. The increase in SO_3 is caused by three properties of the oxy-fuel process whose relative importance is indicated with numbers in Figure 6:

1. The oxidizer in oxy-fuel combustion contains SO_2, which increases the amount of sulphur present during combustion.
2. The oxidizer in oxy-fuel combustion has a higher concentration of O_2, which decreases the volume flow through the furnace and, thus, increases the concentration of SO_3.
3. The change from N_2 to CO_2 increases the SO_3/SO_2 ratio, due to chemical effects discussed below.

In the temperature region corresponding to a residence time between 2 and 2.5 seconds in Figure 6, SO_3 is mainly formed by Reaction 4. In this region no decisive differences in SO_3-formation between air and oxy-fuel combustion is detected. At lower temperatures (between 2.5 and 3 seconds), the secondary formation of SO_3 via $HOSO_2$ (Reactions 5 and 6) is more important. From previous work [31] it is known that the increased concentration of CO_2 during oxy-fuel combustion affects the radical pool, primarily through:

$$CO_2 + H \rightarrow CO + OH \quad (12)$$

The elevated OH-radical concentration enhances secondary formation of SO_3. Wet recycle with higher concentration of H_2O in the furnace increases this effect slightly, leading to a small increase in SO_3 during wet recycling in the calculations, mentioned above. However, in practice, the concentration of SO_2 in the oxidizer is probably lower in wet recycle, and consequently, also the concentration of SO_3. In the calculations the chemical influence of the H_2O concentration is of interest, and therefore the inlet SO_2 concentration is kept the same for dry and wet recycle.

The temperature profile and the stoichiometric ratio were the same in the air and oxy-fuel cases. However, the possibly more intense combustion process during oxy-fuel combustion could promote both lower stoichiometric ratio and faster cooling rate. Figure 7 illustrates the influence of the cooling process and the stoichiometric ratio on the outlet SO_3 concentration in oxy-fuel combustion. The simulation is done for the case of an oxy-fuel flue gas with an initial concentration of 2000 ppm SO_2 and an initial temperature of 1800°C cooled to 400°C. Similar to an air-fired furnace, the cooling rate (or residence time in the crucial temperature range) and the stoichiometric ratio during the cooling are of great importance to SO_3 formation, while the influence of the peak temperature is negligible.

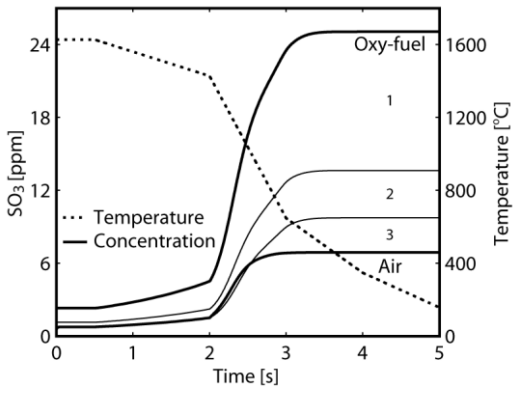

Figure 6. SO_3 concentration during combustion of Lausitz lignite for air and oxy-fuel combustion with dry recycle. The areas 1 to 3 are defined in the text.

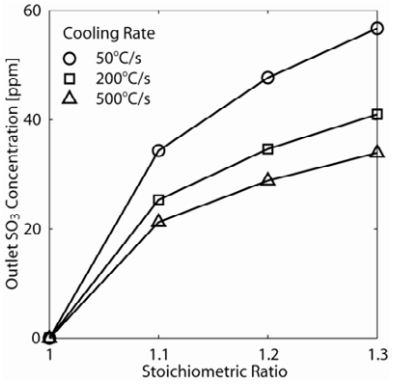

Figure 7. Influence of cooling rate and stoichiometric ratio on SO_3 formation under oxy-fuel conditions (2000 ppm SO_2 initial concentration and an initial temperature of 1800°C cooled to 400°C).

3.3. Acid dew-point

Besides the concentration of SO_3, the concentration of H_2O in the flue gas is of importance for the acid dew-point. Figure 8, valid for atmospheric pressure, shows the acid dew-point temperature as a function of the SO_3 concentration at three water concentrations, corresponding to the cases: air-firing (9% H_2O), oxy-fuel with dry recycle (12% H_2O), and, oxy-fuel with wet recycle (37% H_2O). The acid dew-point temperature increases with the concentration of SO_3 and H_2O in the flue gas. In oxy-fuel combustion the dew-point temperature is higher due to the higher SO_3 concentration. The influence of the H_2O content in the flue gas is considerable in wet flue gas recycle. In this case, an increase in acid dew-point temperature by 20 to 30K is expected due to higher SO_3 and H_2O concentrations.

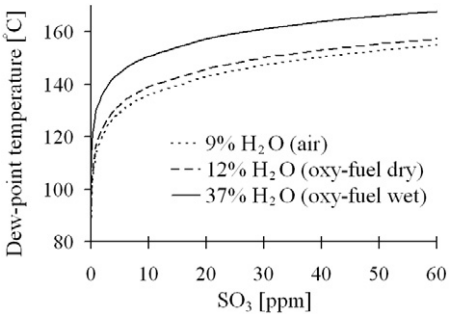

Figure 8. Acid dew-point temperature at three water concentrations (atmospheric pressure).

3.4. Hydrogen sulphide

The possibility of hydrogen sulphide (H_2S) formation during oxy-fuel combustion is illustrated in Figure 9. The concentration of H_2S is calculated in a flame with an initial SO_2 concentration of 1000 ppm. Formation of H_2S can be considerable under sub-stoichiometric conditions ($\lambda<1$) combined with temperatures between 1100°C and 1600°C, i.e. conditions typically occurring in a flame. If oxygen is in excess with quantities typical for pulverized coal combustors, the outlet concentration of H_2S is negligible. An increased H_2S concentration can be anticipated in an oxy-fuel flame, due to the SO_2 content in the oxidizer. Furthermore, the recycle rate in oxy-fuel operation can directly influence the H_2S concentration since the flame temperature has a considerable influence on the formation under sub-stoichiometric conditions (see Figure 9).

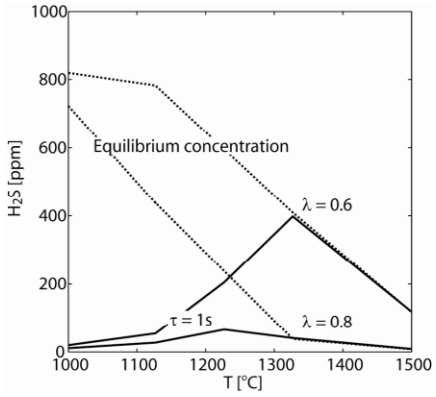

Figure 9. Concentration of hydrogen sulphide at equilibrium (dashed line) and after 1 second reaction time (full line, τ) at the stoichiometric ratios (λ) 0.6 and 0.8 and 1000 ppm initial concentration of SO_2.

4. Conclusions

Laboratory tests of the coal examined in this work (Lausitz lignite) show that the release of SO_2 is strongly dependent on the combustion temperature. In oxy-fuel combustion, the furnace temperature is closely related to the flue gas recycle rate, which also governs the conversion of fuel-S to SO_2. Previous experimental work shows that the conversion of fuel-S to SO_2 is lower in oxy-fuel than in air-fuel conditions. The SO_2 emission per unit energy supplied is therefore reduced. On the other hand, the SO_2 concentration is higher in oxy-fuel combustion due to the recycling of SO_2 and the higher O_2 concentration compared to air-firing. An important reason for the interest in sulphur chemistry is the formation of SO_3/H_2SO_4, which may cause operational problems due to low temperature corrosion. Modelling of the gas-phase chemistry shows that the elevated SO_2 mole fraction results in a higher concentration of SO_3 in the flue gas; about four times higher than in air combustion. Also the reduced flow and the change of the combustion environment from N_2 to CO_2 increase the concentration of SO_3. It is therefore important to further quantify and clarify the formation of SO_3 and its consequences to secure the operation of oxy-fuel power

plants. Hence, measurements of SO_3 and the acid dew-point in the oxy-fuel combustion process are needed to complement the modelling results.

Acknowledgment

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